Practical 1P4

Metallography

SAFETY INFORMATION

1) Wear **lab coat** at all times while in sample preparation lab.
2) Wear **safety glasses and gloves** during grinding and polishing, and during etching.
3) **Etching can only be done under supervision.**
4) DO NOT wear gloves in the microscopes room.
5) Always carry out etching in a **fume cupboard**.
6) While etching, always use **tongs** to handle the specimen.
7) **Etchant splashes on the skin or eyes must be washed with copious amounts of water. Get immediate help from PCT (Diana).**

What you should learn from this practical

Science:

This practical ties-in with the lecture course on Microstructure of Materials. It will help you to understand:

1) How to use a phase diagram to predict the microstructure of a material.
2) The effect of rolling and annealing on the microstructure of ***Cu***.
3) The effect of solidification on the microstructure of a ***single phase Cu-Zn***.
4) The effect of solidification on the microstructure of ***two-phase Al-Cu alloys***.
5) The effect of annealing followed by cooling at different rates on the microstructure of a ***carbon steel***.

Practical skills:

You will learn:

1) How to polish and etch metallographic sections of Cu, Al and Fe based alloys.
2) How to use a reflection optical microscope to investigate microstructures at a range of magnifications.

Overview of practical:

A total of 8 metallographic specimens for examination in a reflection ***optical microscope*** will be prepared, recording the observed microstructures by ***photomicrography***. You will then use the relevant ***phase diagram*** to explain in each case the mechanism of formation of the ***microstructure*** and the ***microstructural features*** observed.

Experimental details

Divide the samples equally so that all of you develop competence in polishing and etching techniques.

Metallographic preparation:

The preparation of each specimen (apart from Al-Cu ones; more on that below) involves ***grinding***, ***polishing*** and ***etching*** in that order. Most of the specimens have been used previously for this experiment, and only need polishing and etching, but new or damaged specimens will need to be ground first. The experimental techniques of grinding, polishing and etching will be shown to you by the senior and/or junior demonstrators. Make sure that you are careful in using the grinders and polishers, and handling the etching acids.

Optical microscopy:

After preparation, each specimen should be ***mounted*** on a glass slide with plasticine, ***levelled***, and then ***examined*** in one of the reflection optical microscopes. The senior and junior demonstrators will show you how to use the microscopes. Always examine specimens at ***low magnification first***, and then with ***progressively higher magnifications***. Much information can be lost by not examining at a low magnification.

Recording the microstructure:

Each member of the group should examine each specimen and record the observed microstructure by taking a microphotograph. Structures should be photographed at a magnification which shows important features clearly. Make sure that you ***record the magnification*** in each case.

Specimens:

There are a total of 8 specimens to be examined, listed as follows:

1) Copper alloys:

A4 Impure copper, chill cast (rapidly cooled in copper mould). Etching: **alcoholic $FeCl_3$ solution**; it produces a faceted finish and distinguish grains clearly. A low magnification will suffice.

A5 As above, after cold rolling and annealing. Etching: **alcoholic $FeCl_3$ solution**. Note the fine regular grain structure and the appearance of annealing twins which were absent in the cast material.

A6 Cu-30wt.%Zn. α-Brass, as cast. Etching: **alcoholic $FeCl_3$ solution**. At low magnifications a coarse grain structure is apparent, but the outlines of grains are ragged – explain why. Within each grain a fine criss-cross pattern of dendrites is to be seen - dendrites being lower in zinc content and relatively bright. Between the dendrites the zinc content is higher and therefore these regions are more heavily etched. Note that although dendrites are all of the same preferred growth orientation, the different sections produce a wide variety of patterns.

2) Aluminium alloys:

A9-11 Al - 25, 33 and 40wt.%Cu. Slow cooled from the melt. **These samples already come prepared – polished and etched**. This is because the etchant Al alloys require – aqueous **1%HF + 1%HCl + 1%HNO_3** – contains dangerous chemicals. Note the various proportions of the primary phases and the finer eutectic structure which is visible at high magnification.

3) Steels:

S6 Fe-0.85wt%C. Three samples in the same mount:

(i) Larger diameter sample - normalised from 1100K;

(ii) Middle sample - as (i) but re-annealed at 1100K for 30 min and furnace cooled;

(iii) Third sample - as (i) but re-annealed at 1220k for 30 min and cooled at 1K per minute to room temperature.

Etching: **nital** (alcohol solution of HNO_3). Initial large grains of γ (austenite) decompose with a eutectic like reaction (called eutectoid - solid solutions) to form plates of α (ferrite) and Fe_3C (cementite). The specific eutectoid reaction at 1000K is:

$$\gamma(0.77\%C) \Leftrightarrow \alpha(0.02\%C) + Fe_3C(6.7\%C)$$

This structure is called pearlite (ferrite and cementite) and is only formed during slow cooling – explain why. Try and determine where the original γ grain boundaries were and how growth of the pearlite is directed relative to these boundaries. Also, look at the rim and at the center of each sample and explain any differences between these regions.

S17 Fe-0.8%C steel quenched into water from 1273K to give martensitic structure which can be revealed by a **relatively heavy etch in nital**.

Etching safety considerations:

Nital: corrosive, toxic and flammable.

Alcoholic solution of $FeCl_3$: corrosive, toxic and flammable.

What should be in the Lab notebook

- Do not take the lab notebooks out of the Teaching labs. Please make sure you have enough time to writeup and keep it short, but relevant.
- Include a very short overview of the practical and describe experimental methods briefly – sample preparation and imaging.
- The annotated sketch or photograph of the microstructure of each specimen should be included in the report, with the magnification indicated at the corner, preferably by a scale marker. Please annotate the micrographs to indicate features that you wish to reveal.
- Give a brief description of the main features of each microstructure.
- The relevant phase diagram for each specimen should be consulted and also **included in the write up**. You may either choose to print these or hand draw the relevant portion. Indicate positions of the investigated specimens in the diagram by an arrow. Use these positions within the phase diagram, as well as knowledge of the thermal treatment of each sample, to explain the differences in the observed microstructures.

References

1) Hansen – “The Constitution of Binary Alloys”.
2) Smithells – “Metals Reference Book”.
3) A.S.M. – “Metals Handbook”.
4) W. Hume-Rothery “The Structure of Alloys of Iron”.
5) V. Voort – “Metallography Principles & Practice”.
6) G.A. Chadwick – “Metallography of Phase Transformations”.
7) Higgins – “Engineering Metallurgy”.